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METHOD FOR THE DETERMINATION OF
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METHOD FOR THE DETERMINATION OF MINERAL MATTER IN COAL

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Indian Standard

METHOD FOR THE DETERMINATION OF MINERAL MATTER IN COAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 September 1967, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 The mineral matter or inorganic constituents of coal are generally the sum of all constituents in coal that are not the part of the organic coal substance that is carbon, hydrogen, nitrogen, oxygen, and sulphur (organic), or moisture associated with coal. In all fundamental studies on coal, correct evaluation of mineral matter is essential as the composition of the coal substance which is calculated from the analysis of the air-dried sample can only be arrived at if the quantity of non-coal substance is accurately known. For low ash coals, difference between the results expressed on dry, ash-free and on dry, mineral-free basis is not very significant; but for high ash coals the difference is appreciable. Therefore, the determination of mineral matter in high-ash coals, as in the case of most Indian coals, is of utmost importance.

0.3 The mineral constituents of coal are intimately bound with the coal substance thus making the separation of mineral matter by any physical method impossible. In this direct method, the coal is partially demineralized, which leaves the coal substance unaffected. By separate estimation of ash and of iron, the mineral matter is calculated. Indirect methods have also been developed, where mineral matter is calculated from the ash content. However, such a method is not covered in this standard.

0.4 This standard is based essentially on the draft ISO/R/679 'Determination of mineral matter in coal' (*revised text*) prepared by Technical Committee ISO/TC 27 Solid Mineral Fuels of the International Organization for Standardization.

0.5 In reporting the result of an analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the direct method for the determination of mineral matter in coal.

2. PRINCIPLE

2.1 The coal sample is partially demineralized by treatment with hydrochloric and hydrofluoric acids under such conditions that the coal substance remains unaffected. The loss in mass of the coal due to the acid treatment is recorded and the insoluble part of the mineral matter determined by ashing the partially demineralized coal. In addition, the iron content of the ash is determined so that the pyrites present in the extracted coal can be calculated. The amount of hydrochloric acid absorbed by the coal substance is also determined.

3. APPARATUS

3.1 All the apparatus listed below should be made from material resistant to acids, especially hydrofluoric acid. A suitable material is polyvinyl chloride (PVC) or high density polyolefins.

3.1.1 Beaker — a 200-ml beaker with a cover slip.

3.1.2 Thermometer Pocket — tube, sealed at one end to carry a thermometer.

3.1.3 Stirrer

3.1.4 Wash-bottle

3.1.5 Filter — with a sintered alumina filter plate as shown in Fig. 1.

3.1.6 Filter Flask

3.1.7 Balance — sensitive to 0.1 mg.

4. REAGENTS

4.1 Hydrochloric Acid — relative density, 1.155, conforming to IS : 265-1962*.

4.2 Hydrochloric Acid — approximately 5 N.

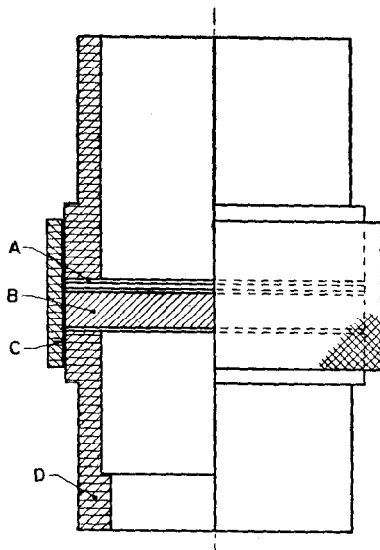
4.3 Hydrofluoric Acid — relative density, 1.13 or 1.14.

5. PROCEDURE

5.1 Mix the air-dried sample of coal ground to pass a 212-micron

*Specification for hydrochloric acid (*revised*).

IS Sieve thoroughly for at least one minute, preferably by mechanical means. Alternatively, the coal sample may be dried at 105° to 110°C.



- A — Paper filter, connected to the upper cylinder with an adhesive substance
- B — Plate of sintered alumina
- C — Rubber ring
- D — Cylinder of acid-resistant material (PVC)

FIG. 1 FILTERING DEVICE

5.2 Weigh accurately about 6 g of the sample into beaker and add 40 ml of the hydrochloric acid, 5 N. For low rank and other reactive coals, the acids may be placed in the beaker before adding the coal sample to avoid local over-heating. Insert the stirrer and the tube carrying a thermometer and place the cover slip over the beaker. Place the beaker in a water bath, maintained at 55° to 60°C. Stir the contents at 5 minutes intervals, remove the beaker after 45 minutes and allow the coal suspension to settle for 10 minutes. Decant the solution through the filter under suction. Wash any coal on the filter with water, drain and transfer the coal back to the beaker with the aid of not more than 5 ml of water. Care is required to avoid the loss of coal by splashing (see Note). Add 40 ml of the hydrofluoric acid to the beaker and repeat the heat treatment and filtration as previously described. Rinse any coal on the filter back into the beaker with not more than 5 ml water. Add 50 ml of 5N hydrochloric acid to the

beaker, replace it in the water bath and repeat the heat treatment previously described. Decant the solution through the prepared filtering device and wash the coal with water three times, decanting each time. Transfer the coal entirely to the filter and wash 20 times with 25 ml portions of hot water each time. Remove any residual coal from the beaker by means of a rubber tipped rod and cold water. Drain the coal under suction for 5 to 10 minutes.

NOTE — The first hydrochloric acid extraction is unnecessary for coals having carbon dioxide content of less than 0.5 percent.

5.3 Dismantle the filter, break up the compacted, wet coal and dry the filter top and cool in a vacuum oven at 50°C and a pressure of 25 mm Hg for about 90 minutes. Remove and allow to cool in air to attain equilibrium until constant weight is obtained. Generally an hour's cooling shall be required. Recover the coal and transfer as much as possible to a glass stoppered bottle. Wipe the filter top and filter paper free from coal and reweigh. Obtain the weight of extracted coal by difference.

5.4 Mix the extracted coal thoroughly and determine its moisture and ash according to 6 and 8 of IS:1350-1959* respectively and chlorine by the Eschka method described in 7 of IS:1352-1959†, as well as the total iron content of the ash in accordance with IS:1355-1959‡; determine also the moisture content of the original sample, according to IS:1350-1959*. Calculate the hydrochloric acid equivalent to the chlorine content and the pyrites equivalent to the total iron content.

6. CALCULATION AND REPORTING OF RESULTS

6.1 All results should be quoted on a moisture-free basis; an example of the calculation is given in Appendix A.

$$\begin{array}{l} \text{Percentage of} \\ \text{mineral mat-} \\ \text{ter, } MM \end{array} = \frac{m_1 - m_2 + P + \text{HCl} + 1.1 A}{m_1} \times 100 \text{ (see Note)}$$

$$\text{and } F = \frac{MM}{A_1}$$

where

m_1 = mass in grams of sample taken,

m_2 = mass in grams of sample after extraction,

*Methods of test for coal and coke — proximate analysis, total sulphur and chlorine value.

†Methods of test for coal and coke — special impurities.

‡Methods of test for ash of coal and coke.

- P = mass in grams of pyrites in the extracted coal,
 HCl = mass in grams of hydrochloric acid in the extracted coal,
 A = mass in grams of ash, less iron oxide from the pyrites in the extracted coal,
 F = mineral matter factor, and
 A_1 = percentage of ash in the original coal.

NOTE — The factor 1.1 allows approximately for the water of hydration of the aluminium and silicon compounds in the demineralized coal. In most cases this correction is small and can be ignored.

6.2 The result (preferably the mean of duplicate determinations) should be reported to the nearest 0.1 percent.

APPENDIX A

(Clause 6.1)

EXAMPLE FOR METHOD OF CALCULATION

Original coal (dry)		Extracted coal:	
6.836 g		Hydrochloric acid (dry basis)	1.06 per-cent
Treated coal (dry)		Ash (dry basis)	6.03 per-cent
3.205 g			
Loss in mass		Iron oxide (dry basis)	4.2 per-cent
3.631 g		Ash-iron oxide (residual ash)	1.83 per-cent
			0.034 g
			0.135 g
			0.058 g
Loss in mass	3.631 g		
Hydrochloric acid	0.034 g		
Pyrites	0.207 g		
Residual ash $\times 1.1$	0.064 g		
Mass of mineral matter	3.936 g		
Mineral matter (dry basis)	57.6 percent		
Ash content of original coal (dry basis)	52.8 percent		
Mineral matter factor = $\frac{\text{mineral matter}}{\text{ash}} = \frac{57.6}{52.8} = 1.09$			